

Supporting Information

For

Three-Dimensional Architectures for Highly Stable Pure Blue Emission

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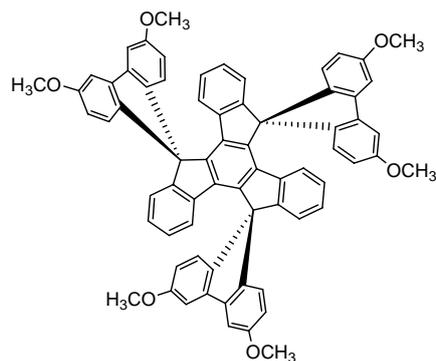
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1. Experimental section

General Methods. Chemicals were purchased and used as received. 2-Bromo-3',5-dimethoxy-1,1'-biphenyl, **4** and **5** were prepared following literature procedures.^{1,2} All air and water sensitive reactions were performed under nitrogen atmosphere. Toluene and tetrahydrofuran (THF) were distilled from sodium and benzophenone ketyl. ¹H and ¹³C NMR spectra were recorded on a Varian Mercury 200 MHz or Mercury plus 300 MHz using CDCl₃ as solvent unless otherwise noted. All chemical shifts were reported in parts per million (ppm), ¹H NMR chemical shifts were referenced to TMS (0 ppm) or CHCl₃ (7.26 ppm), and ¹³C NMR chemical shifts were referenced to CDCl₃ (77.00 ppm). Absorption spectra were recorded on PerkinElmer Lambda 35 UV-*vis* Spectrometer. Photoluminescent (PL) spectra were carried out on PerkinElmer LS55 Luminescence Spectrometer. MALDI-TOF mass spectra were recorded on a Bruker BIFLEX III or AUTOFLEX III time-of-flight (TOF) mass spectrometer (Bruker Daltonics, Billerica, MA, USA) using a 337 nm nitrogen laser with dithranol as matrix. Elemental analyses were performed using a German Vario EL III elemental analyzer. Thermal gravity analyses (TGA) were carried out on a TA Instrument Q600 analyzer and differential scanning calorimetry (DSC) analyses were performed on a METTLER TOLEDO Instrument DSC822^e calorimeter. Cyclic voltammetry was performed using BASI Epsilon workstation and measurements were carried out in acetonitrile containing 0.1 M *n*Bu₄NPF₆ as a supporting electrolyte. Carbon electrode was used as a working electrode and a platinum wire as a counter electrode; all potentials were recorded versus Ag/AgCl

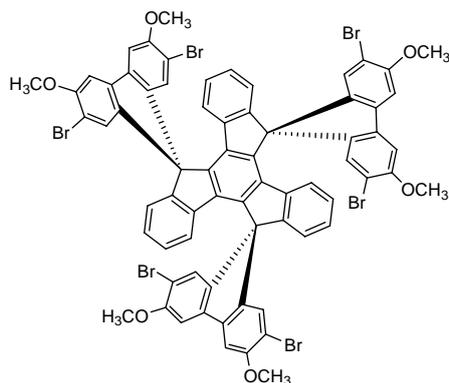
(saturated) as a reference electrode. The scan rate was 100 mV s⁻¹.

Device Fabrication and Characterization. LED was fabricated on pre-patterned indium-tin oxide (ITO) with sheet resistance 10–20 Ω/square. The substrate was ultrasonic cleaned with acetone, detergent, deionized water, and 2-propanol. Oxygen plasma treatment was made for 10 min as the final step of substrate cleaning to improve the contact angle just before film coating. Onto the ITO glass was spin-coated a layer of PEDOT:PSS film with thickness of 50 nm from its aqueous dispersion, aiming to improve the hole injection and to avoid the possibility of leakage. PEDOT:PSS film was dried at 80 °C for 2 h in the vacuum oven. The solution of all materials in *p*-xylene was prepared in a nitrogen-filled drybox and spin-coated on top of the ITO/PEDOT:PSS surface. Typical thickness of the emitting layer was 50-80 nm. Then a thin layer of barium as an electron injection cathode and the subsequent 200 nm thick aluminum protection layers were thermally deposited by vacuum evaporation through a mask at a base pressure below 2×10^{-4} Pa. The deposition speed and the thickness of the barium and aluminum layers were monitored by a thickness/rate meter. The cathode area defines the active area of the device. The typical active area of the devices in this study is 0.15 cm². The EL layer spin coating process and the device performance tests were carried out within a glovebox with nitrogen circulation. The luminance of the device was measured with a calibrated photodiode. External quantum efficiency was verified by the measurement of the integrating sphere, and luminance was calibrated after the encapsulation of devices with UV-curing epoxy and thin cover glass.



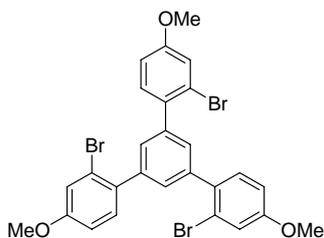
2c. To a well-degassed solution of 2-bromo-3',5-dimethoxy-1,1'-biphenyl (2.93 g, 10 mmol) in THF (40 mL) was added *t*-BuLi (13.3 mL, 20 mmol, 1.5 M in pentane) at -78°C . The mixture was stirred for 1 h, and then a suspension of truxenone (576 mg, 1.5 mmol) in toluene (10 mL) was added. After continuously stirring at room temperature for 3 h, the mixture was quenched with saturated NH_4Cl . The aqueous layer was extracted with ethyl acetate. The combined extracts were dried over MgSO_4 . After removal of solvents under vacuum, the residue was purified by column chromatography over silica gel (eluent: petroleum ether (PE):EtOAc = 5:1) to afford triol as a yellow solid. After the resulting triol was dissolved in refluxing CH_3COOH (10 mL), $\text{CH}_3\text{SO}_3\text{H}$ (0.2 mL) was injected to the solution. The mixture was refluxed for 2 h. The product as a slightly yellow powder was filtered off and washed with ethanol. Yield: 39%. Small amount of constitutional isomers was observed.³ ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 7.47-7.46 (d, $J = 2.4$ Hz, 6H, Ar-H), 6.78-6.75 (6H, d, $J = 8.4$ Hz, Ar-H), 6.76-6.74 (3H, m, Ar-H), 6.69-6.64 (6H, d, $J = 8.4$ Hz, $J = 2.4$ Hz, Ar-H), 6.65-6.62 (3H, m, Ar-H), 6.54-6.52 (3H, d, $J = 7.2$ Hz, Ar-H), 6.34-6.31 (3H, d, $J = 7.2$ Hz, Ar-H), 3.88 (18H, s, CH_3). ^{13}C NMR (CDCl_3 , 75 MHz, ppm): δ 159.8, 150.8, 142.9, 141.3, 141.0, 138.0, 127.0, 126.8, 126.4, 124.3, 124.1, 122.2, 114.2, 105.8, 65.5, 55.5. MALDI-TOF MS: Calcd for $\text{C}_{69}\text{H}_{48}\text{O}_6$: 972.3. Found: 972 (M^+).

Anal. Calcd for C₆₉H₄₈O₆: C, 85.16; H, 4.97. Found: C, 84.66; H, 4.74.



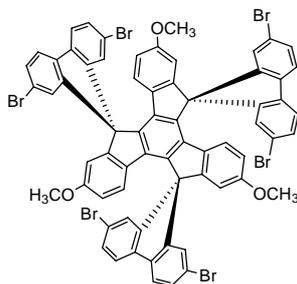
2a. To a solution of **2c** (214 mg, 0.206 mmol) in CH₂Cl₂ (60 mL) was added a solution of bromine (250 mg, 1.24 mmol) in CH₂Cl₂ (0.5 mL). The mixture was stirred for 20 h. Saturated aqueous NaHSO₃ solution was added to remove excess bromine. Removal of solvents under vacuum afforded the crude product as a slightly yellow solid. The powder was dissolved in CH₂Cl₂, and then precipitated by addition of ethanol. After filtration, the desired product was collected as a slightly yellow powder. Compound **2a** was directly used in the following Suzuki coupling without further purification. Yield: 94%. ¹H NMR (CDCl₃, 300 MHz, ppm): δ 7.45 (6H, s, Ar-H), 7.02 (6H, s, Ar-H), 6.87-6.84 (3H, m, Ar-H), 6.73-6.70 (3H, m, Ar-H), 6.52-6.49 (3H, d, *J* = 7.8 Hz, Ar-H), 6.35-6.32 (3H, d, *J* = 7.8 Hz), 4.09 (18H, s, CH₃).

MALDI-TOF MS: Calcd for C₆₉H₄₂Br₆O₆: 1445.8. Found: 1446 (M⁺).



3. To a solution of 2-bromo-4-methoxyacetophenone (3.50 g, 15.3 mmol) in ethanol (50 mL) was added dropwise SiCl₄ (7.0 mL) under the ice bath. The mixture was

stirred at room temperature for 15 h and then 50 mL of CH₂Cl₂ was added. The aqueous layer was extracted with CH₂Cl₂. The combined extracts were dried over MgSO₄. After removal of solvents under reduced pressure, the residue was purified via chromatography over silica gel (eluent: PE:CH₂Cl₂ = 5:1) to give the product as a white solid. Yield: 63%. ¹H NMR (CDCl₃, 300 MHz, ppm): δ 7.43 (s, 3H, Ar-H), 7.38-7.35 (d, *J* = 8.7 Hz, 3H, Ar-H), 7.24-7.23 (d, *J* = 2.7 Hz, 3H, Ar-H), 6.94-6.91 (dd, *J* = 8.7 Hz, *J* = 2.7 Hz, 3H, Ar-H), 3.84 (s, 9H, CH₃). ¹³C NMR (CDCl₃, 50 MHz, ppm): δ 159.3, 139.9, 134.4, 131.9, 129.7, 122.8, 118.3, 113.5, 55.4. EI-MS, *m/z*: 634.0 (M⁺+2, 100%). Anal. Calcd for C₂₇H₂₁Br₃O₃: C, 51.22; H, 3.34. Found: C, 51.38; H, 3.49.

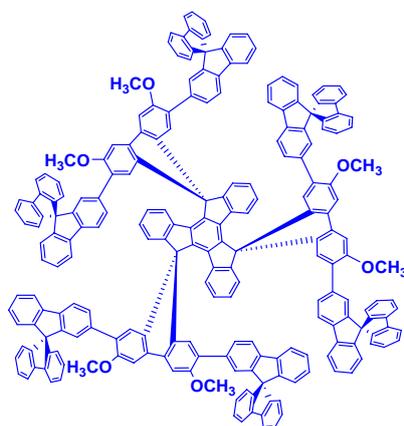


2b. To a solution of **3** (1.0 g, 1.6 mmol) in dry THF was added dropwise *t*-BuLi (9.6 mmol, 6.4 mL, 1.5 M in pentane) under nitrogen atmosphere at -78 °C. The mixture was stirred at -78 °C for 1 h. A suspension of 2,7-dibromofluorenone (1.9 g, 5.8 mmol) in anhydrous THF (10 mL) was added, and then the mixture was allowed to warm up to 50 °C for 2 hours. The mixture was quenched by saturated aqueous NHCl₄ and extracted with ethyl acetate. After removal of the solvents, the residue was purified via chromatography over silica gel (PE:EtOAc = 5:1) to give triol as a yellow solid. Triol was dissolved in CH₂Cl₂ (10 mL), and then BF₃•Et₂O (0.1 mL) was injected at

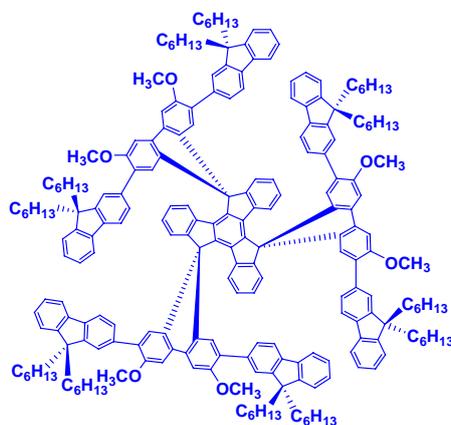
room temperature. The reaction mixture was kept for about 30 min. After filtration, the product was obtained as a white solid. Yield: 32%. ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 7.82-7.79 (d, $J = 8.1$ Hz, 6H, Ar-H), 7.56-7.52 (dd, $J = 8.1$ Hz, $J = 1.8$ Hz, 6H, Ar-H), 7.00-6.99 (d, $J = 1.8$ Hz, 6H, Ar-H), 6.33-6.30 (d, $J = 8.7$ Hz, 3H, Ar-H), 6.23-6.20 (dd, $J = 8.7$ Hz, $J = 2.7$ Hz, 3H, Ar-H), 5.84-5.83 (d, $J = 2.7$ Hz, 3H, Ar-H), 3.50 (s, 9H, CH_3). ^{13}C NMR (CDCl_3 , 75 MHz, ppm): δ 159.3, 150.3, 149.7, 141.5, 139.0, 135.7, 131.2, 130.4, 126.9, 124.8, 123.0, 121.4, 112.2, 108.5, 65.9, 54.7. MALDI-TOF MS: Calcd for $\text{C}_{66}\text{H}_{36}\text{Br}_6\text{O}_3$: 1355.8. Found: 1356 (M^+). Anal. Calcd for $\text{C}_{66}\text{H}_{36}\text{Br}_6\text{O}_3$: C, 58.44; H, 2.68; Br, 35.34. Found: C, 58.50; H, 2.82.

General Suzuki coupling procedure for preparation of 1a-d

To a well-degassed solution of **2a** or **2b** (0.1 mmol) in THF (20 mL) was added 9,9'-spirobifluorenyl-2-boronic ester (309 mg, 0.7 mmol) or 9,9'-dihexylfluorenyl-2-boronic acid (265 mg, 0.7 mmol), NaOH (2 M, 3.0 mL), $\text{Pd}(\text{PPh}_3)_4$ (50 mg) under nitrogen atmosphere. The mixture was refluxed for 30h. The organic layer was separated and washed with saturated aqueous NH_4Cl . After evaporation of the solvents, the residue was purified via chromatography over silica gel (PE: CH_2Cl_2 : EtOAc = 7:1:1) to give pure products as white powders.

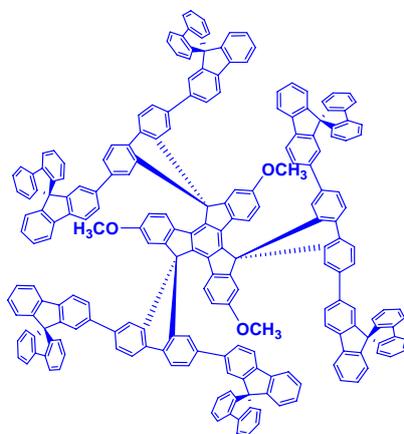


1a. Pure product was obtained as a white powder. Yield: 78%. ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 7.83-7.79 (m, 12H, Ar-H), 7.40-7.38 (d, 6H, $J = 8.0$ Hz, Ar-H), 7.36-7.33 (d, 6H, $J = 8.0$ Hz, Ar-H), 7.30-7.28 (m, 6H, Ar-H), 7.27 (s, 6H, Ar-H), 7.06-6.93 (m, 36H, Ar-H), 6.84 (s, 6H, Ar-H), 6.82-6.81 (d, 6H, $J = 1.6$ Hz, Ar-H), 6.77-6.73 (m, 18H, Ar-H), 6.63-6.58 (m, 3H, Ar-H), 6.54-6.52 (d, 3H, $J = 7.6$ Hz, Ar-H), 6.48-6.43 (m, 3H, Ar-H), 6.25-6.22 (d, 3H, $J = 7.6$ Hz, Ar-H), 3.51 (s, 18H, CH_3). ^{13}C NMR (CDCl_3 , 75 MHz, ppm): δ 156.7, 150.2, 149.2, 148.8, 148.7, 147.8, 142.1, 141.8, 141.63, 141.56, 141.2, 140.1, 137.9, 137.7, 130.6, 128.4, 128.2, 127.8, 127.7, 127.5, 127.4, 126.9, 126.4, 126.1, 124.5, 124.1, 122.1, 120.4, 120.1, 119.8, 115.8, 103.1, 65.8, 55.5. MALDI-TOF MS: Calcd for $\text{C}_{219}\text{H}_{132}\text{O}_6$: 2859.0. Found: 2859 (M^+). MALDI-TOF HRMS: Calcd for $\text{C}_{219}\text{H}_{132}\text{O}_6$: 2859.0091. Found: 2858.9693. Anal. Calcd for $\text{C}_{219}\text{H}_{240}\text{O}_6$: C, 91.99; H, 4.65. Found: C, 91.36; H, 4.73.



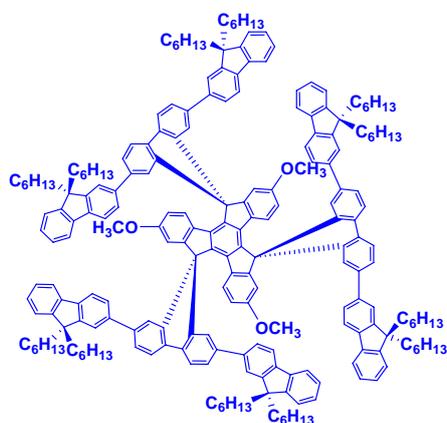
1c. Pure product was obtained as a white solid. Yield: 69%. ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 7.70 (s, 6H, Ar-H), 7.60 (s, 6H, Ar-H), 7.27-7.20 (m, 12H, Ar-H), 7.11 (s, 6H, Ar-H), 7.08-7.06 (d, $J = 7.8$ Hz, 6H, Ar-H), 6.88-6.78 (m, 18H, Ar-H), 6.73-6.69 (m, 6H, Ar-H), 6.67-6.64 (d, $J = 7.5$ Hz, 6H, Ar-H), 3.94 (s, 18H, CH_3), 1.89-1.83 (m, 24H, CH_2), 1.06-1.00 (m, 36H, CH_2), 0.85-0.76 (m, 24H, CH_2),

0.73-0.68 (m, 18H, CH₂), 0.65-0.62 (m, 36H, CH₂), 0.45-0.40 (t, *J* = 7.5 Hz, 18H, CH₃). ¹³C NMR (CDCl₃, 75 MHz, ppm): δ 157.2, 150.8, 150.4, 149.4, 142.2, 141.6, 141.5, 140.7, 140.6, 139.8, 138.4, 136.6, 131.5, 127.6, 127.0, 126.4, 125.0, 124.9, 124.8, 122.4, 120.7, 119.7, 103.8, 65.9, 56.2, 54.7, 40.5, 40.3, 31.4, 31.0, 29.8, 29.5, 23.8, 23.5, 22.6, 22.3, 14.0, 13.7. MALDI-TOF MS: Calcd. for C₂₁₉H₂₄₀O₆: 2967.9. Found: 2968 (M⁺). Anal. Calcd for C₂₁₉H₂₄₀O₆: C, 88.62; H, 8.15. Found: C, 88.55; H, 8.27.



1b. Pure product was obtained as a white solid. Yield: 81%. ¹H NMR (CDCl₃, 300 MHz, ppm): δ 7.87-7.82 (m, 12H, Ar-H), 7.80-7.77 (d, *J* = 8.1 Hz, 6H, Ar-H), 7.63-7.60 (d, *J* = 8.1 Hz, 6H, Ar-H), 7.43-7.40 (d, *J* = 7.5 Hz, 6H, Ar-H), 7.37-7.34 (d, *J* = 7.5 Hz, 6H, Ar-H), 7.31-7.29 (d, *J* = 7.5 Hz, 12H, Ar-H), 7.21 (s, 6H, Ar-H), 7.10-6.97 (m, 36H, Ar-H), 6.79-6.76 (d, *J* = 7.5 Hz, 6H, Ar-H), 6.74-6.71 (d, *J* = 7.5 Hz, 12H, Ar-H), 6.40-6.37 (d, *J* = 8.7 Hz, 3H, Ar-H), 6.03-5.99 (dd, *J* = 8.7 Hz, *J* = 2.4 Hz, 3H, Ar-H), 5.81-5.80 (d, *J* = 2.4 Hz, 3H, Ar-H), 3.28 (s, 9H, CH₃). ¹³C NMR (CDCl₃, 75 MHz, ppm): δ 158.9, 151.9, 149.6, 149.5, 149.2, 148.8, 148.7, 141.8, 141.7, 141.6, 141.04, 140.96, 140.8, 140.6, 140.5, 137.2, 131.1, 128.1, 127.9, 127.8, 127.7, 127.3, 126.8, 125.1, 124.2, 124.1, 124.0, 122.5, 121.5, 120.5, 120.4, 120.3,

120.0, 112.0, 108.3, 66.7, 66.05, 66.02, 54.8. MALDI-TOF MS: Calcd for $C_{216}H_{126}O_3$: 2769.0. Found: 2769 (M^+). Anal. Calcd for $C_{216}H_{126}O_3$: C, 93.68; H, 4.59. Found: C, 93.21; H, 4.63.



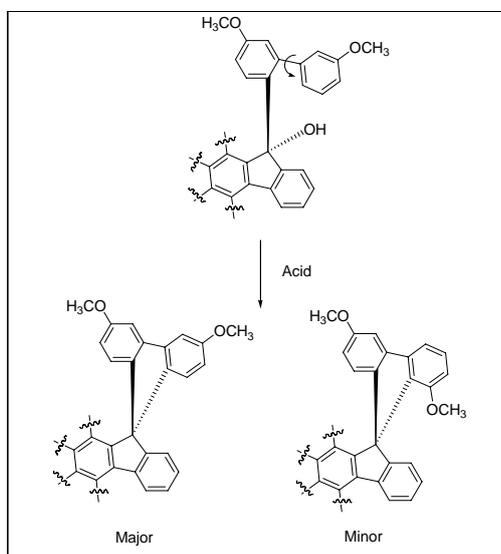
1d. Pure product was obtained as a white solid. Yield: 84%. 1H NMR ($CDCl_3$, 300 MHz, ppm): δ 8.11-8.08 (d, $J = 8.1$ Hz, 6H, Ar-H), 7.71-7.68 (dd, $J = 8.1$ Hz, $J = 1.2$ Hz, 6H, Ar-H), 7.57 (s, 6H, Ar-H), 7.42 (s, 6H, Ar-H), 7.30-7.19 (m, 24H, Ar-H), 7.04-7.02 (d, $J = 7.2$ Hz, 6H, Ar-H), 6.91-6.88 (m, 6H, Ar-H), 6.65-6.62 (d, $J = 8.4$ Hz, 3H, Ar-H), 6.22-6.19 (dd, $J = 8.4$ Hz, $J = 2.4$ Hz, 3H, Ar-H), 6.10-6.09 (d, $J = 2.4$ Hz, 3H, Ar-H), 3.46 (s, 9H, CH₃), 1.95-1.93 (m, 24H, CH₂), 1.07-1.00 (m, 36H, CH₂), 0.89-0.82 (m, 24H, CH₂), 0.80-0.59 (m, 54H, CH₂), 0.53-0.45 (t, $J = 7.2$ Hz, 18H, CH₃). ^{13}C NMR ($CDCl_3$, 75 MHz, ppm): δ 159.0, 152.3, 151.4, 150.7, 149.9, 141.8, 141.6, 140.5, 140.44, 140.41, 139.8, 137.7, 131.6, 127.4, 127.0, 126.7, 125.8, 125.3, 122.6, 121.8, 121.1, 120.7, 120.6, 120.2, 111.5, 108.9, 66.9, 55.1, 55.0, 40.5, 40.3, 31.2, 31.0, 29.53, 29.50, 23.7, 23.6, 22.5, 22.3, 13.9, 13.7. MALDI-TOF MS: Calcd for $C_{216}H_{234}O_3$: 2877.8. Found: 2878 (M^+). Anal. Calcd for $C_{216}H_{234}O_3$: C, 90.14; H, 8.19. Found: C, 89.96; H, 8.22.

References:

- (1) Fournier, J.-H.; Maris, T.; Wuest, J. D. *J. Org. Chem.* **2004**, *69*, 1762-1775.
- (2) Wong, K.-T.; Liao, Y.-L.; Lin, Y.-T.; Su, H.-C.; Wu, C.-c. *Org. Lett.* **2005**, *7*, 5131.
- (3) Due to the rotation of the single bond and consequently the substitution of the ethoxy group at either C3- or C4 position of the phenyl rings, the Friedel-Crafts cyclization of the triol could afford multiple constitutional isomers. The existence of such isomers in similar reaction has been observed in an independent report (Lee, H.; Oh, J.; Chu, H. Y.; Lee, J.-I.; Kim, S. H.; Yang, Y. S.; Kim, G. H.; Do, L.-M.; Z, T.; Lee, J.; Park, Y. *Tetrahedron* **2003**, *59*, 2773-2779). As a result, we have observed multiple signals from the methyl group and corresponding aromatic signals (with relatively low resolution). Fortunately, these isomers did not affect the following reaction and we can remove them by repeated recrystallizations. Nonetheless, we realize that the isomers should not affect their elemental analyses and have acquired these necessary data to confirm their purity. In addition, the bromination of **2c** did not afford pure desired **2a** due to undesired bromination at truxene moiety as a minor side-reaction, and we directly used crude **2a** as a reactant in the following Suzuki coupling reaction. Hence, there is no elemental analysis data of **2a**. Moreover, the solubility of **2a** is very poor in common solvents, and thus ^{13}C NMR spectrum was not recorded. But the structure of the final product **1a** has been unambiguously confirmed by ^1H , and ^{13}C NMR, MS and elemental analysis characterization.

To overcome these problems we developed molecule **1b** as a substitute. As can be

seen from the modified route, there are no isomers formed and we successfully introduced six bromosubstitutions without employing any harsh conditions.



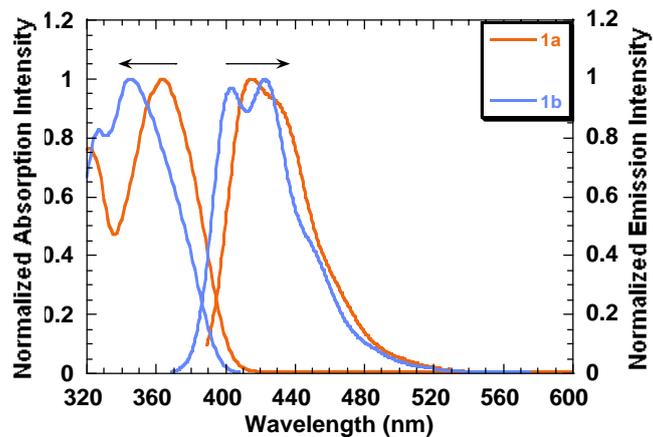


Figure S1. UV-vis and PL spectra of **1a** and **1b** in dilute toluene solution

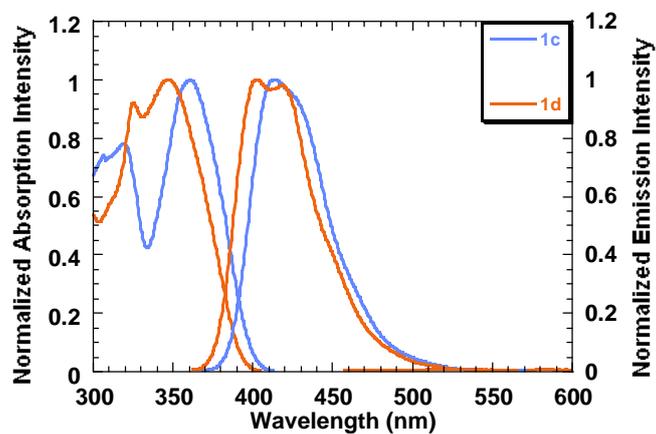


Figure S2. UV-vis and PL spectra of **1c** and **1d** in dilute toluene solution.

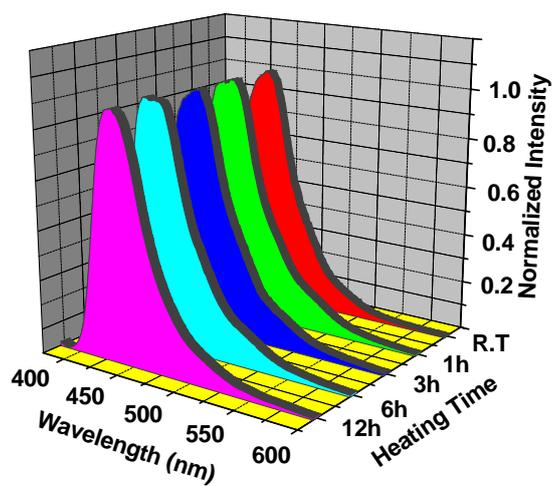


Figure S3. Thermal aging on PL spectra of **1a** in air and under ambient light.

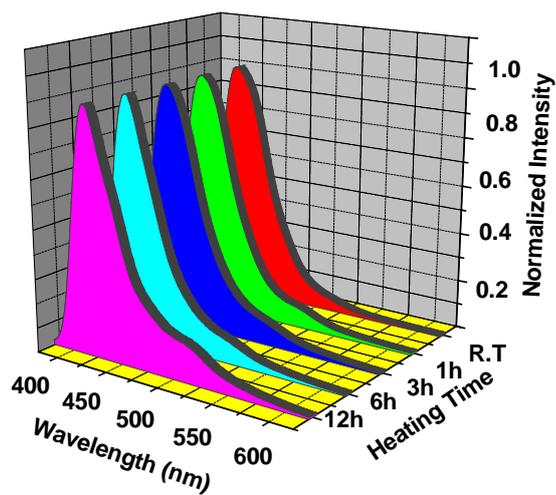


Figure S4. Thermal aging on PL spectra of **1c** in air and under ambient light.

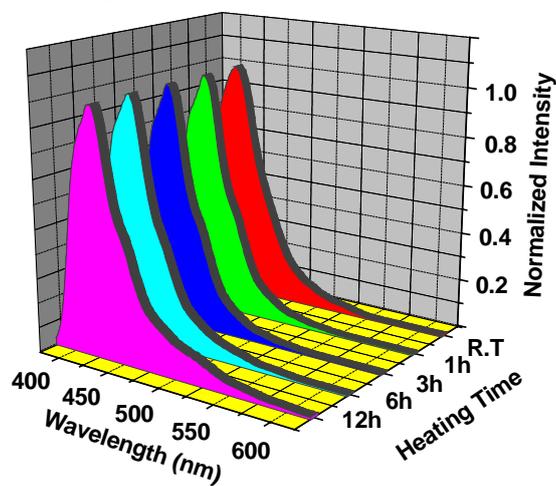


Figure S5. Thermal aging on PL spectra of **1d** in air and under ambient light.

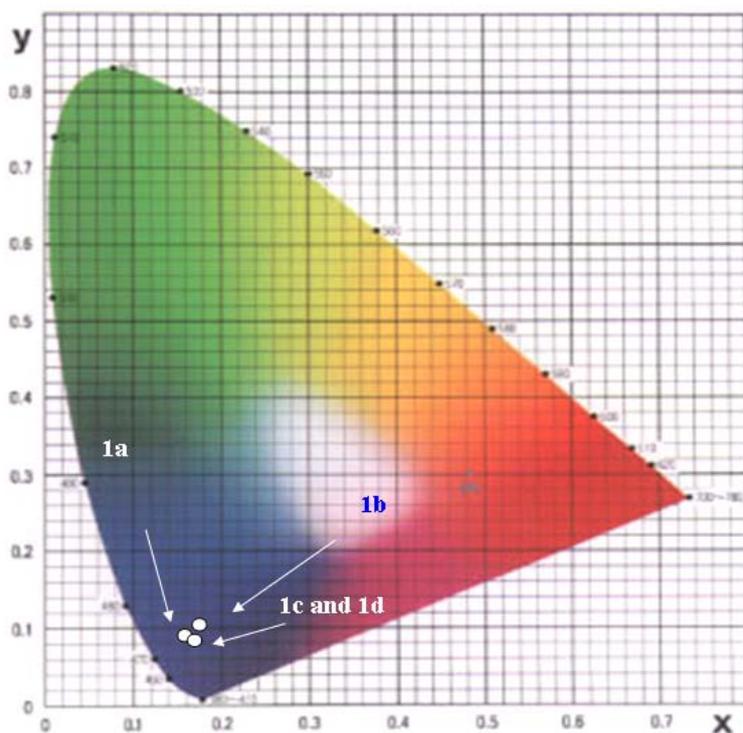


Figure S6. CIE 1931 coordinates of **1a** and **1b**

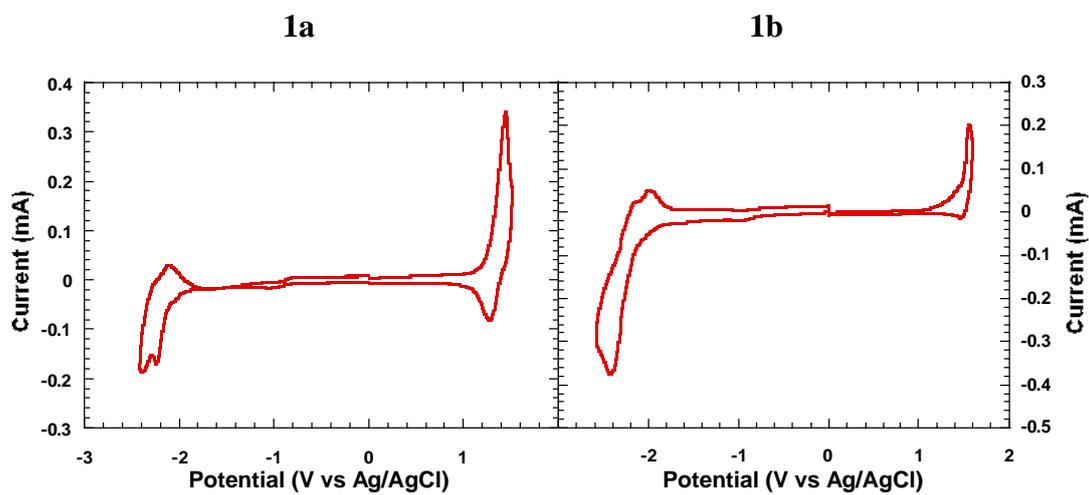


Figure S7. Cyclic voltammograms of **1a** and **1b** in thin films.

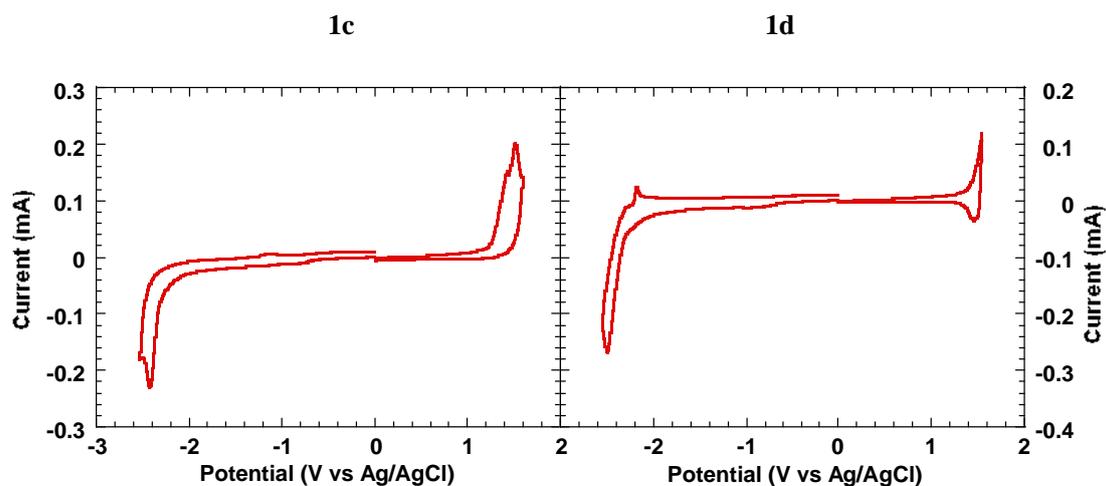


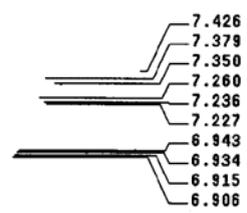
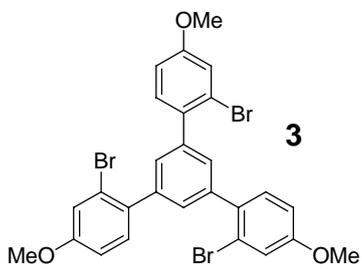
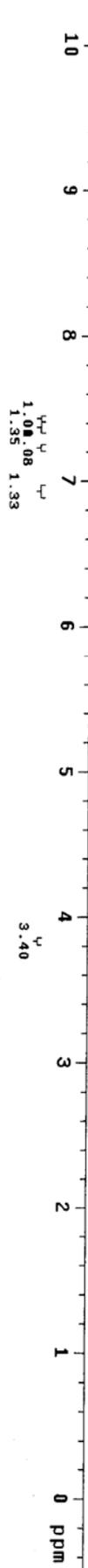
Figure S8. Cyclic voltammograms of **1c** and **1d** in thin films.

Table S1. The photophysical and electrochemical properties of **1a**, **1b**, **1c** and **1d**.

Compd	Abs ^a λ_{\max} , nm (log ϵ)	PL ^a λ_{\max} , nm	Abs ^b λ_{\max} , nm	PL ^b λ_{\max} , nm	Φ_f^c	Φ_f^d	HOMO (eV) ^e	LUMO (eV) ^e	Bandgap (eV) ^f	Bandgap (eV) ^g
1a	364 (5.57)	415	362	439	32%	35%	-5.50	-2.48	3.02	3.04
1b	345 (5.79)	403, 422	347	429	36%	39%	-5.56	-2.50	3.06	3.10
1c	361 (5.62)	413	361	419	51%	54%	-5.51	-2.39	3.12	3.06
1d	347 (5.30)	419	325	416	57%	67%	-5.63	-2.47	3.16	3.14

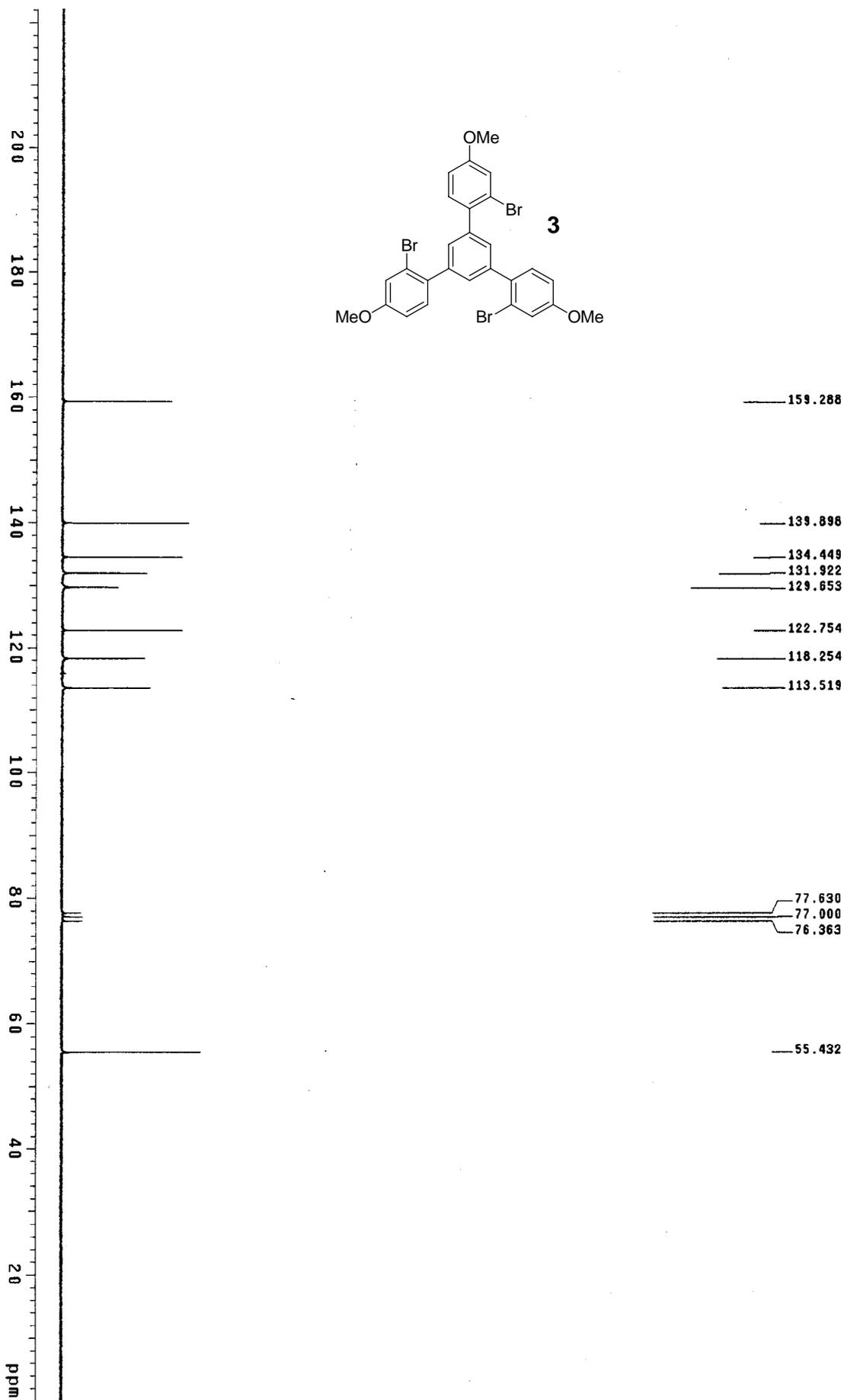
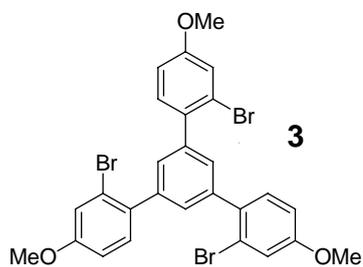
^a Measured in dilute solution (10^{-6} M); ^b Measured in thin films spin-cast from toluene solution (20 mg/mL); ^c Measured in pristine films spin-cast from toluene solution (20 mg/mL); ^d Measured in films annealed for 2 h under nitrogen atmosphere; ^e Cyclic voltammetry analyses were performed in thin films; ^f Calculated based on cyclic voltammetry results; ^g Calculated based on absorption spectra.

trimer



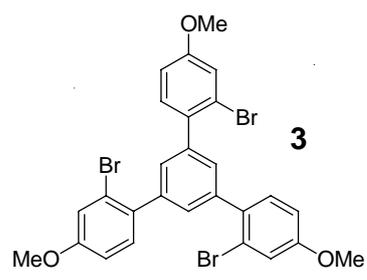
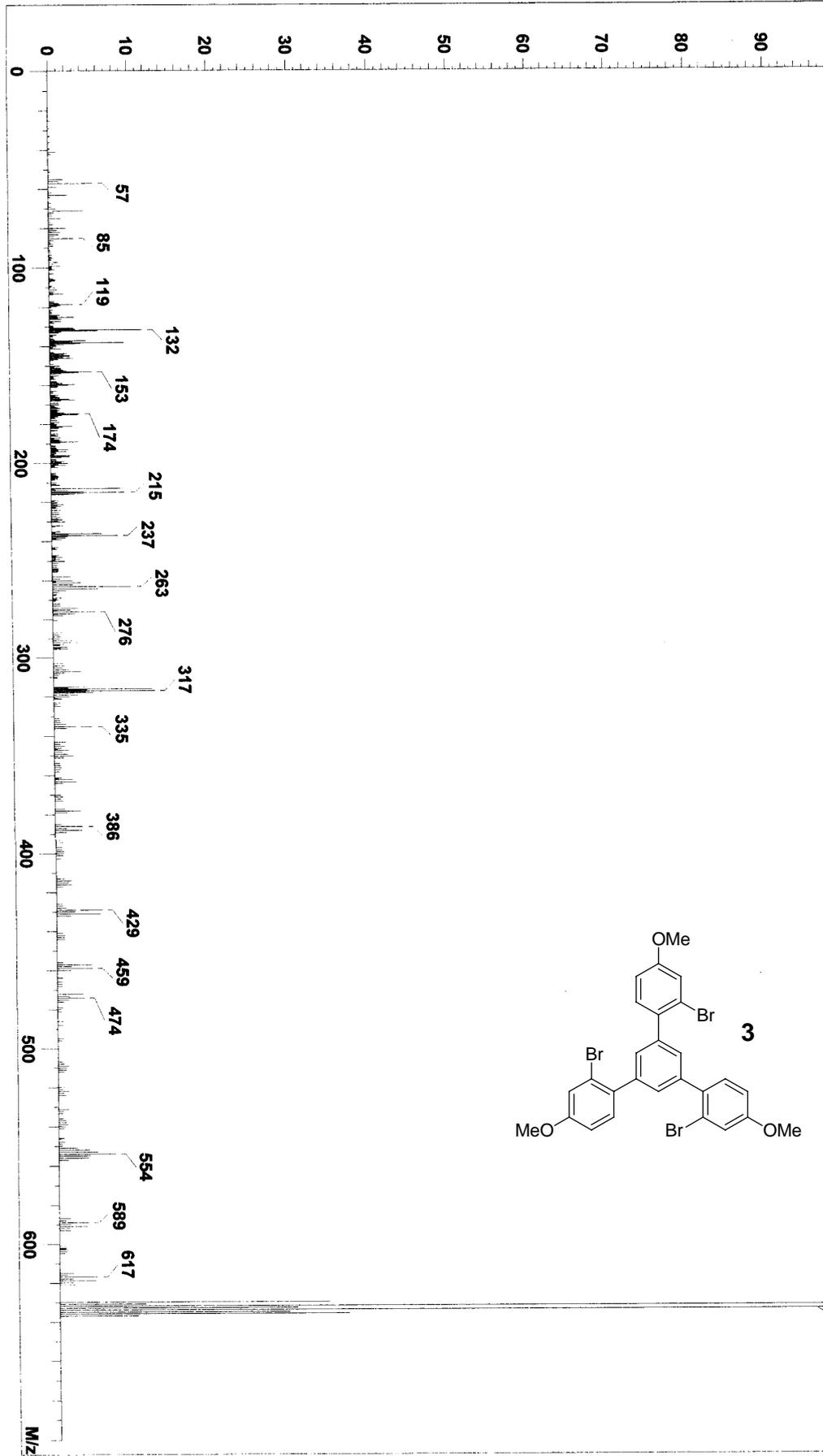
3.836

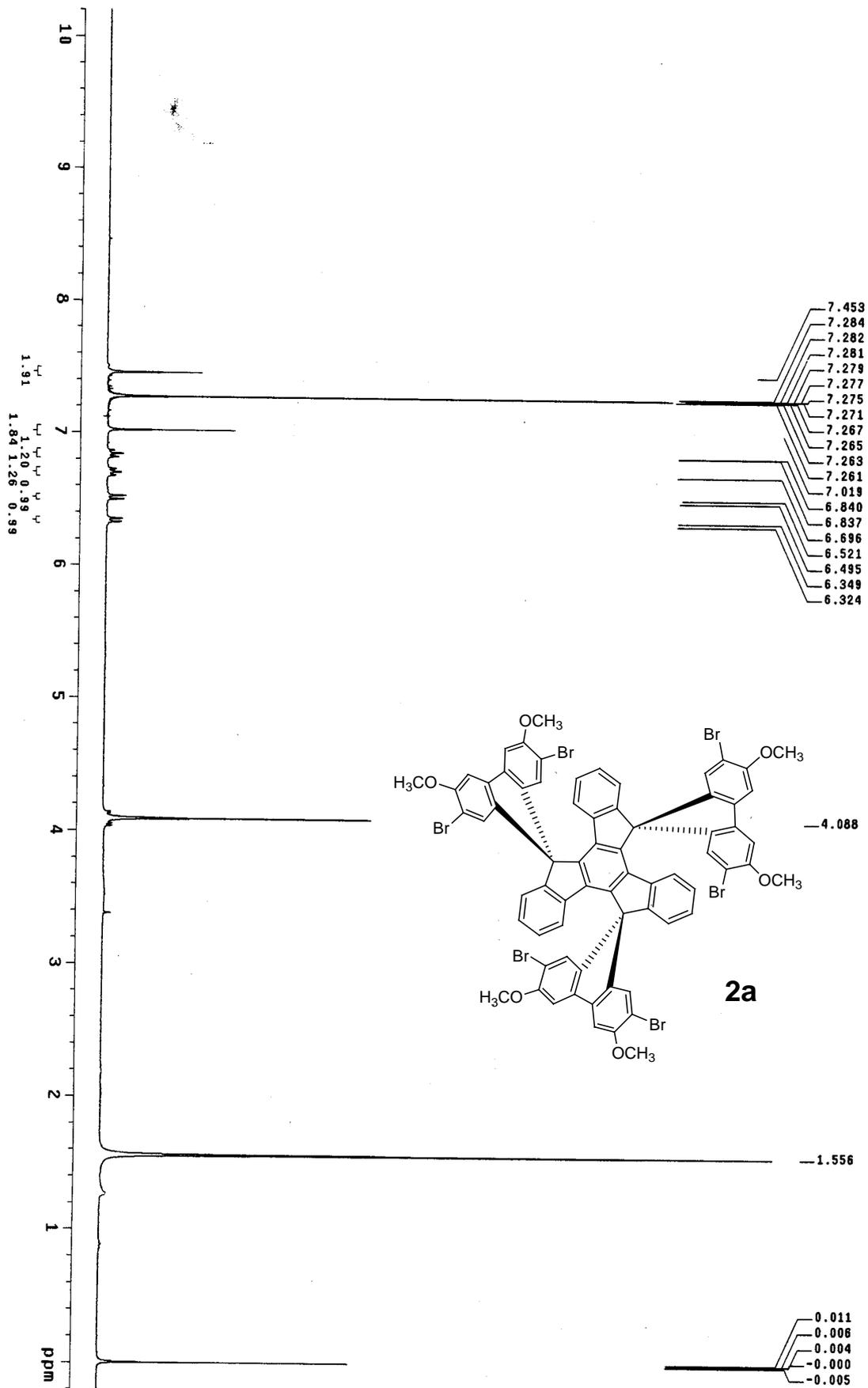
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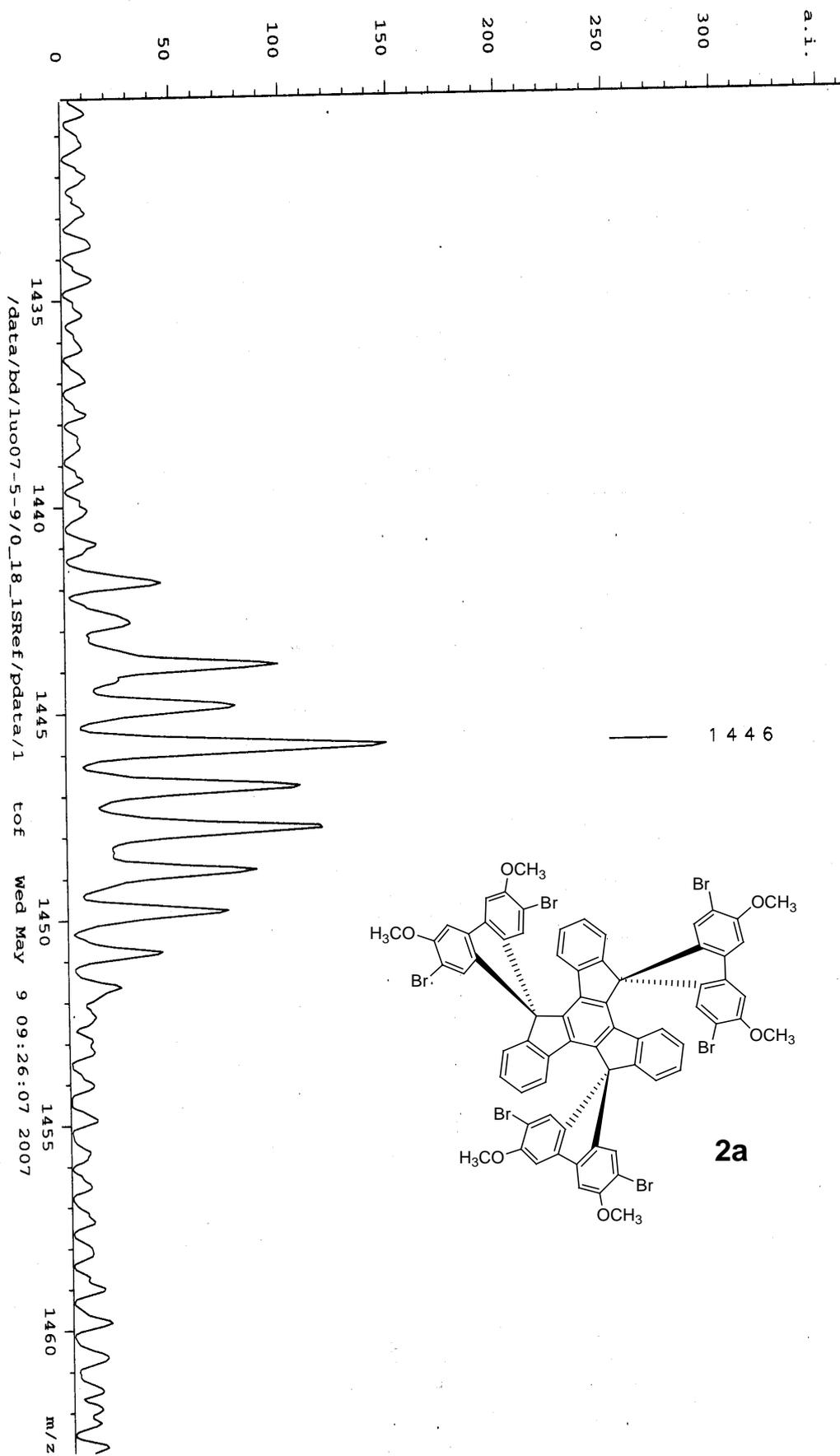


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 File Source : Acquired on MASPec II system [msw/9888]
 Operator : Peking University
 Instrument : ZAB-HS

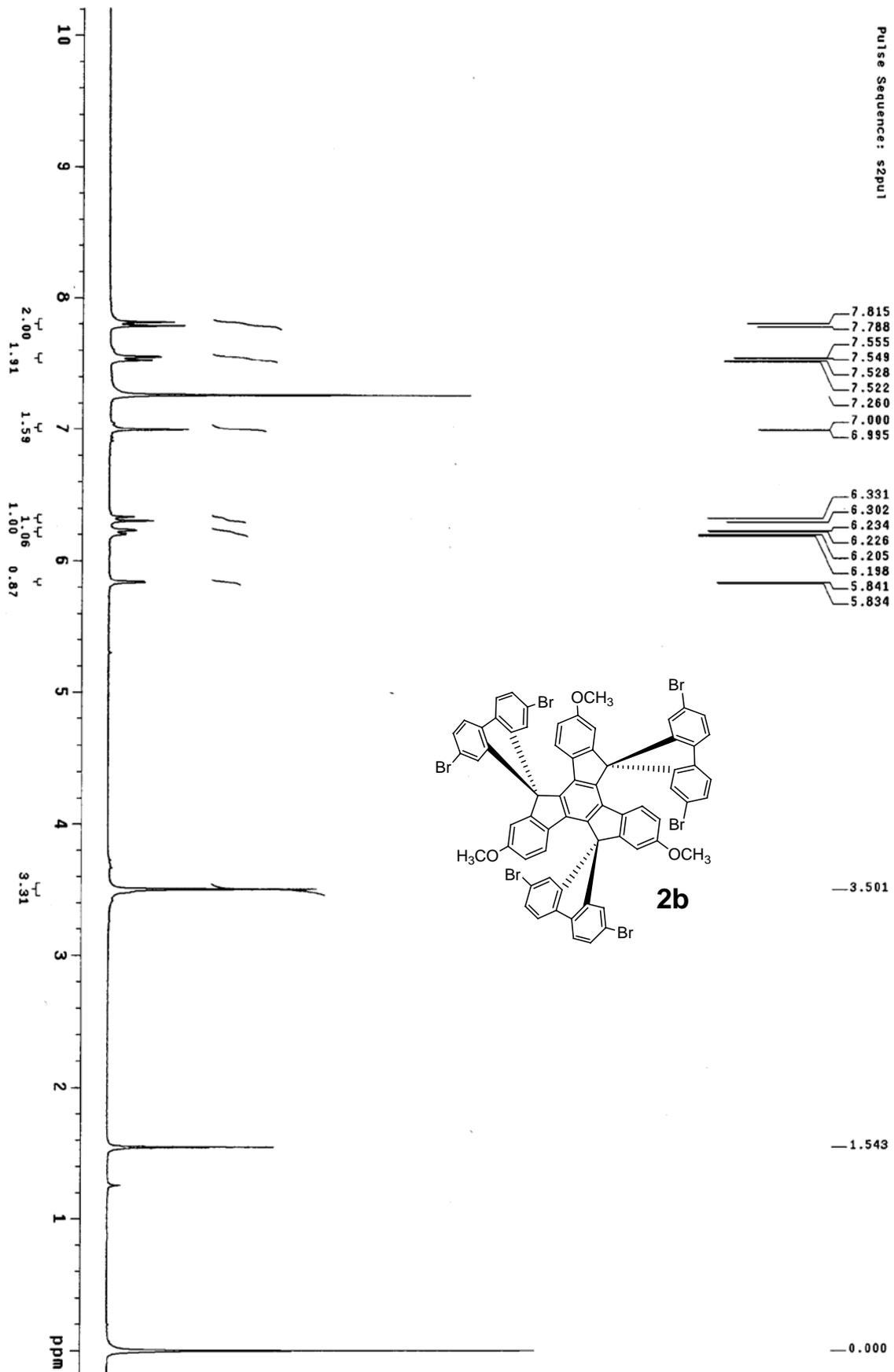
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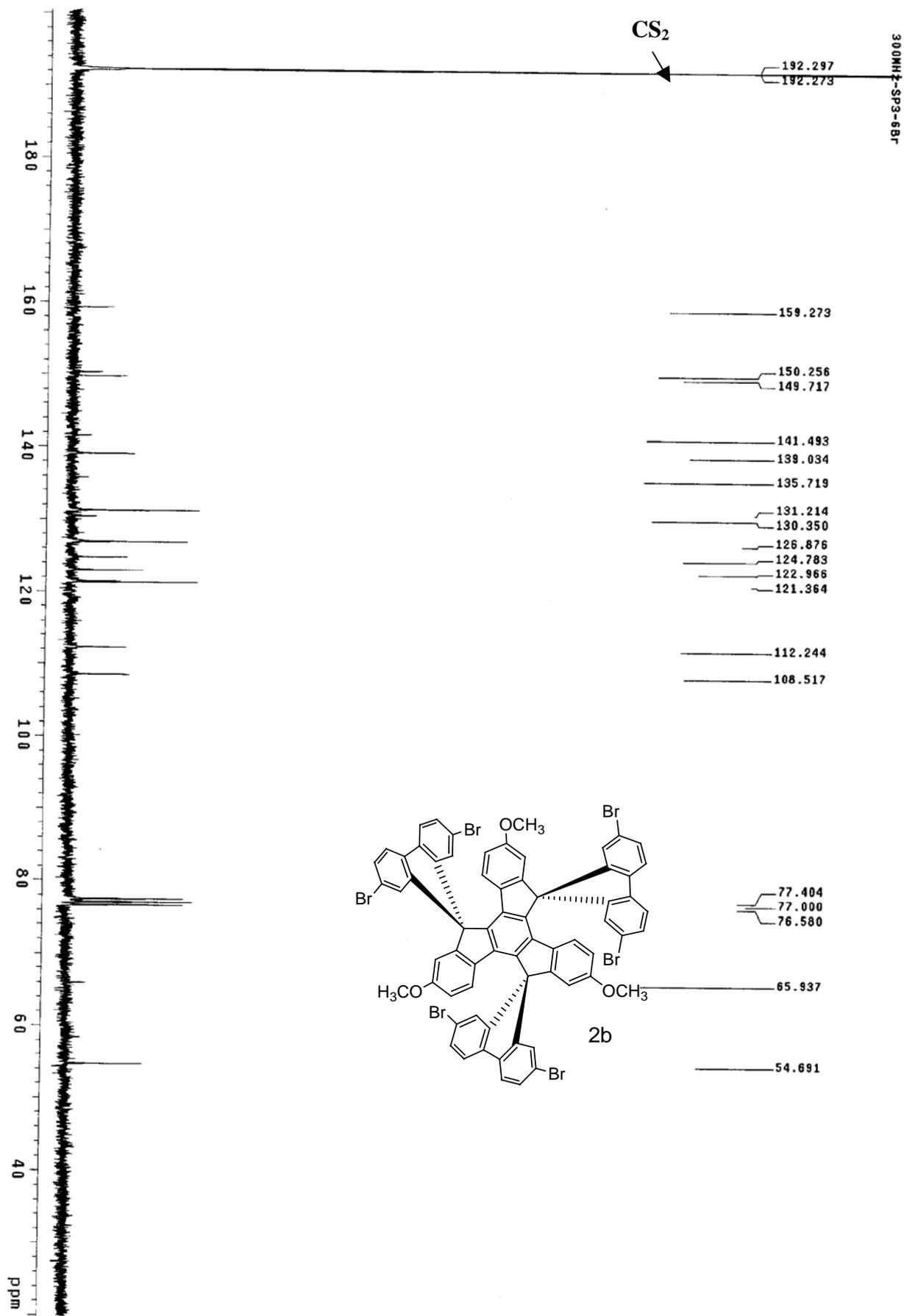




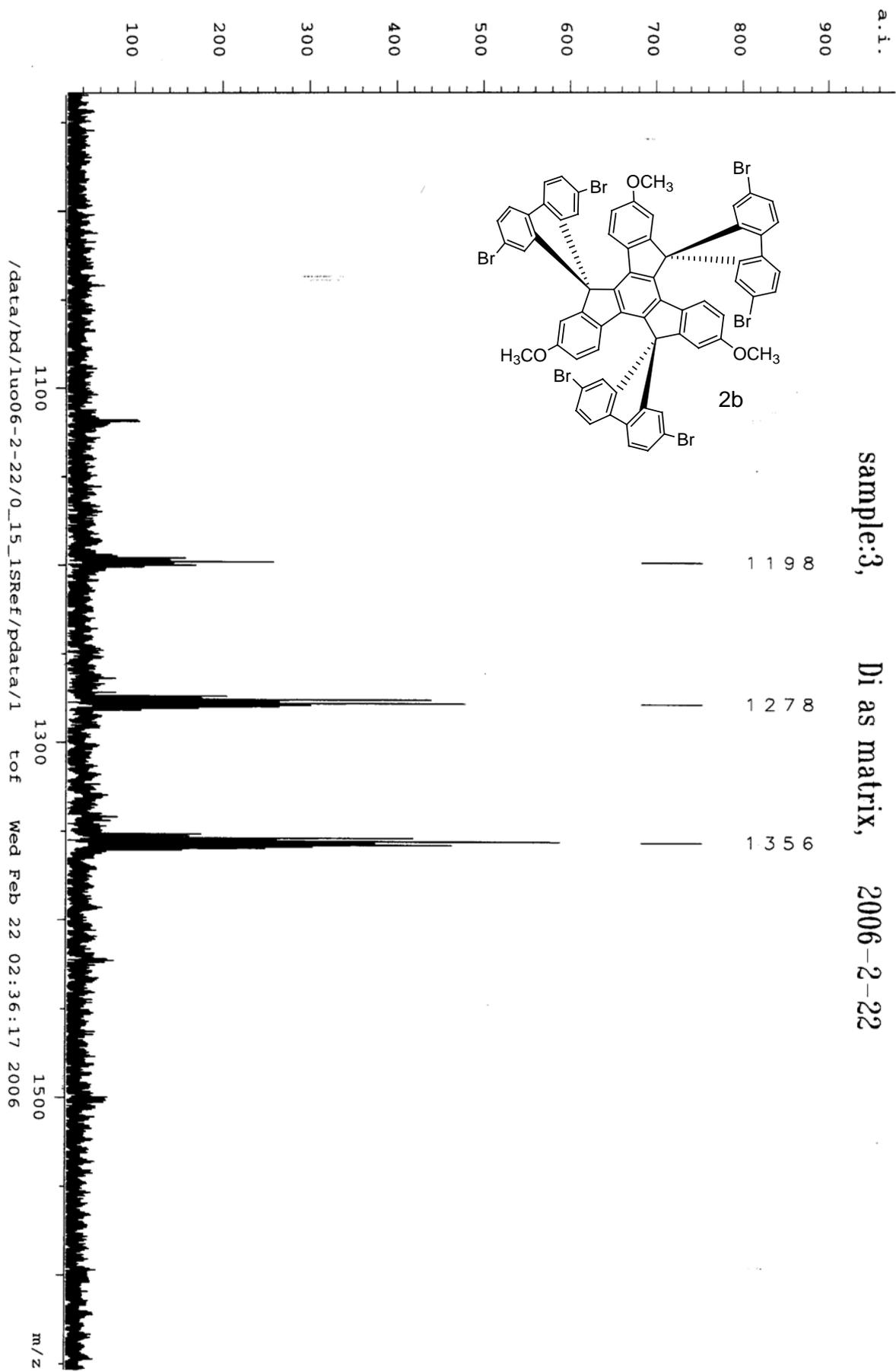


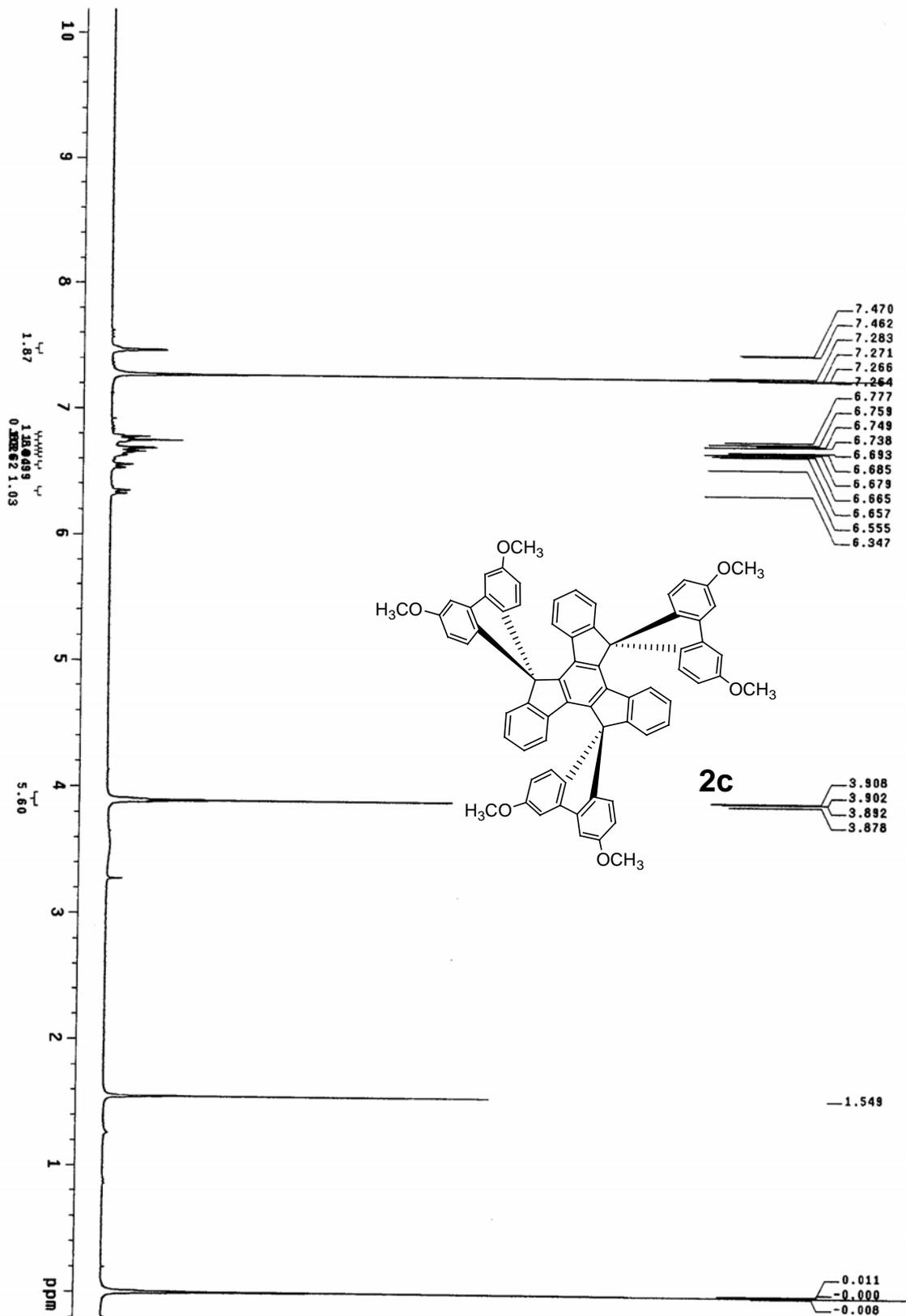
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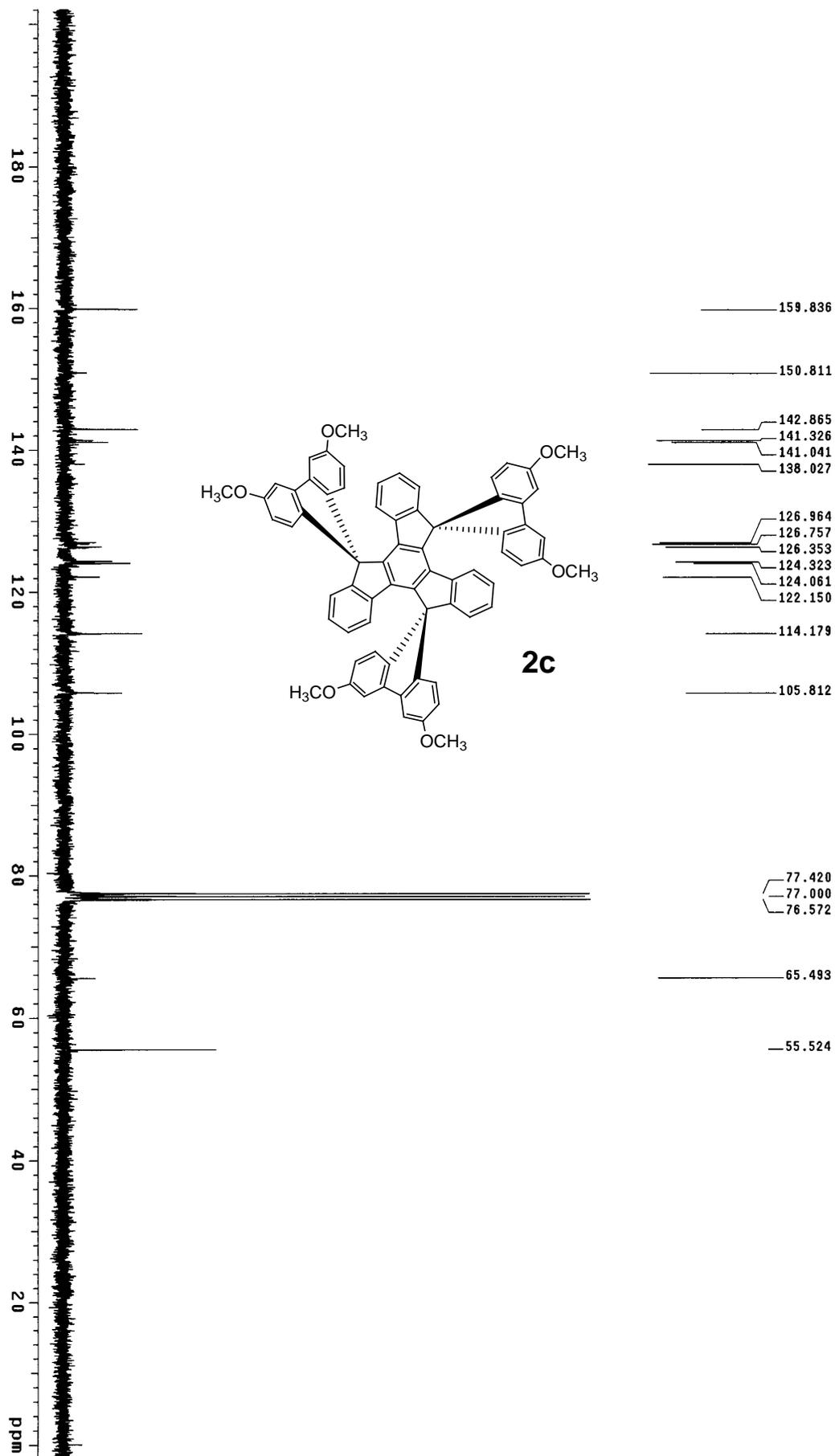


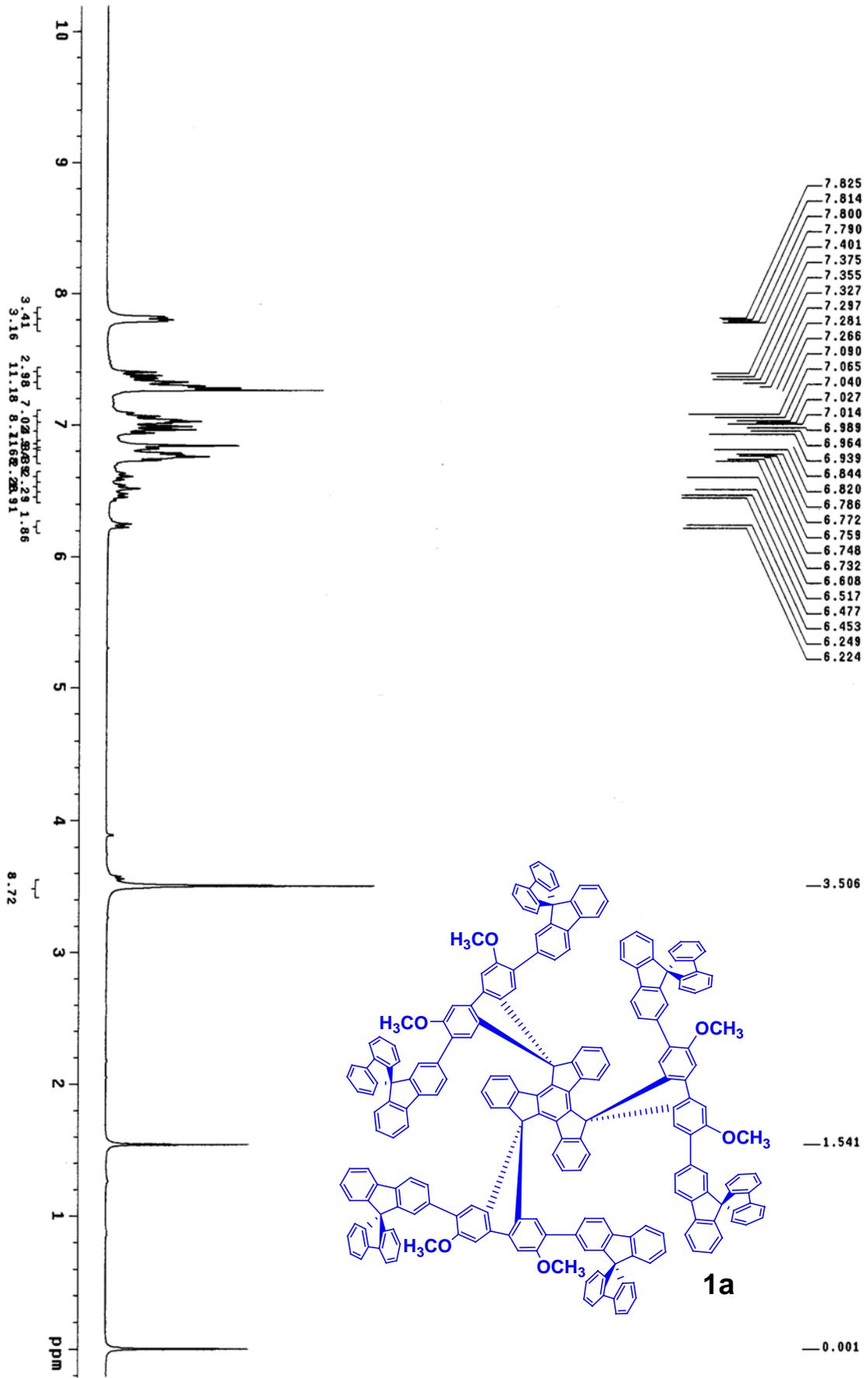


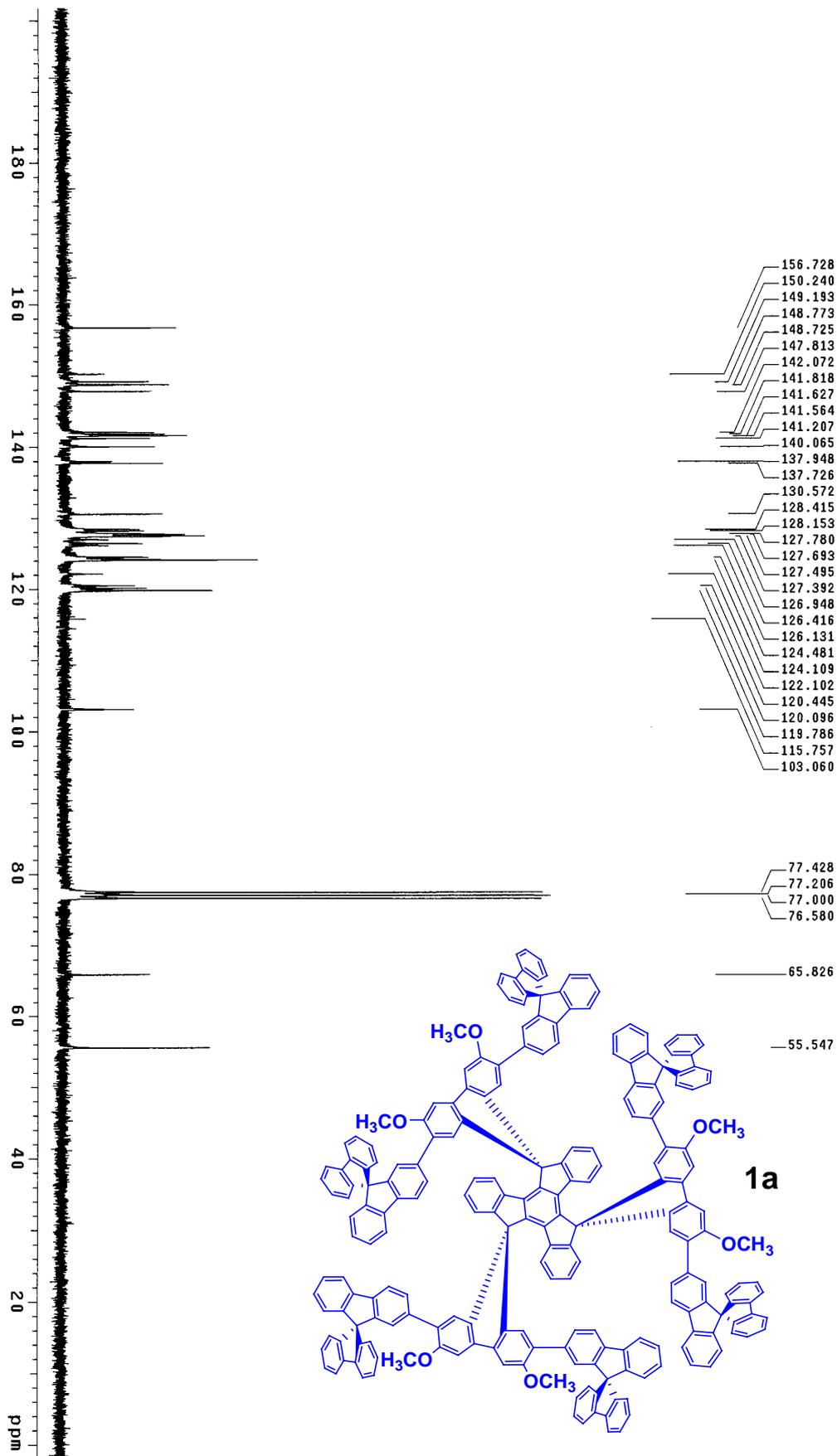
sample:3, Di as matrix, 2006-2-22



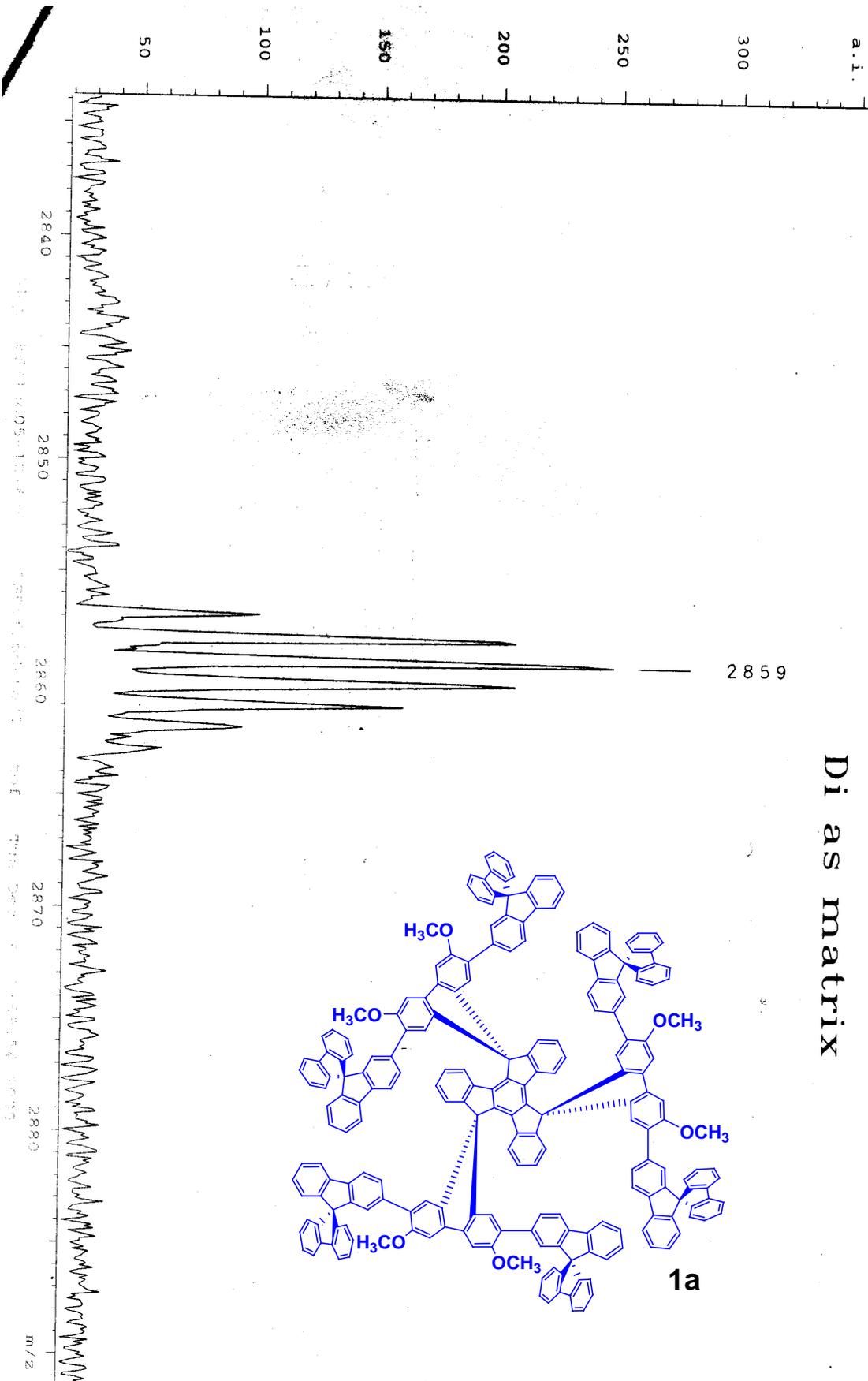




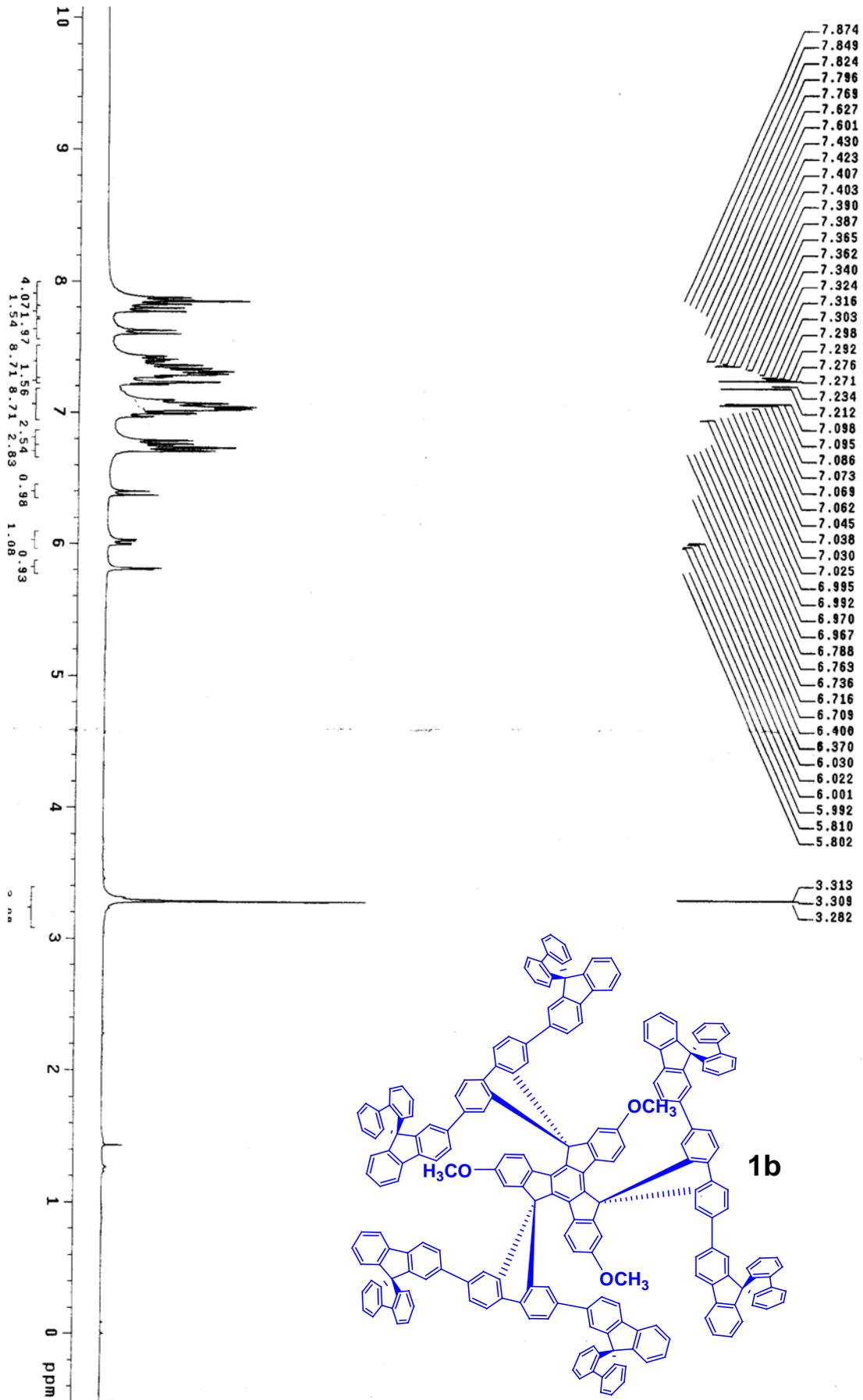


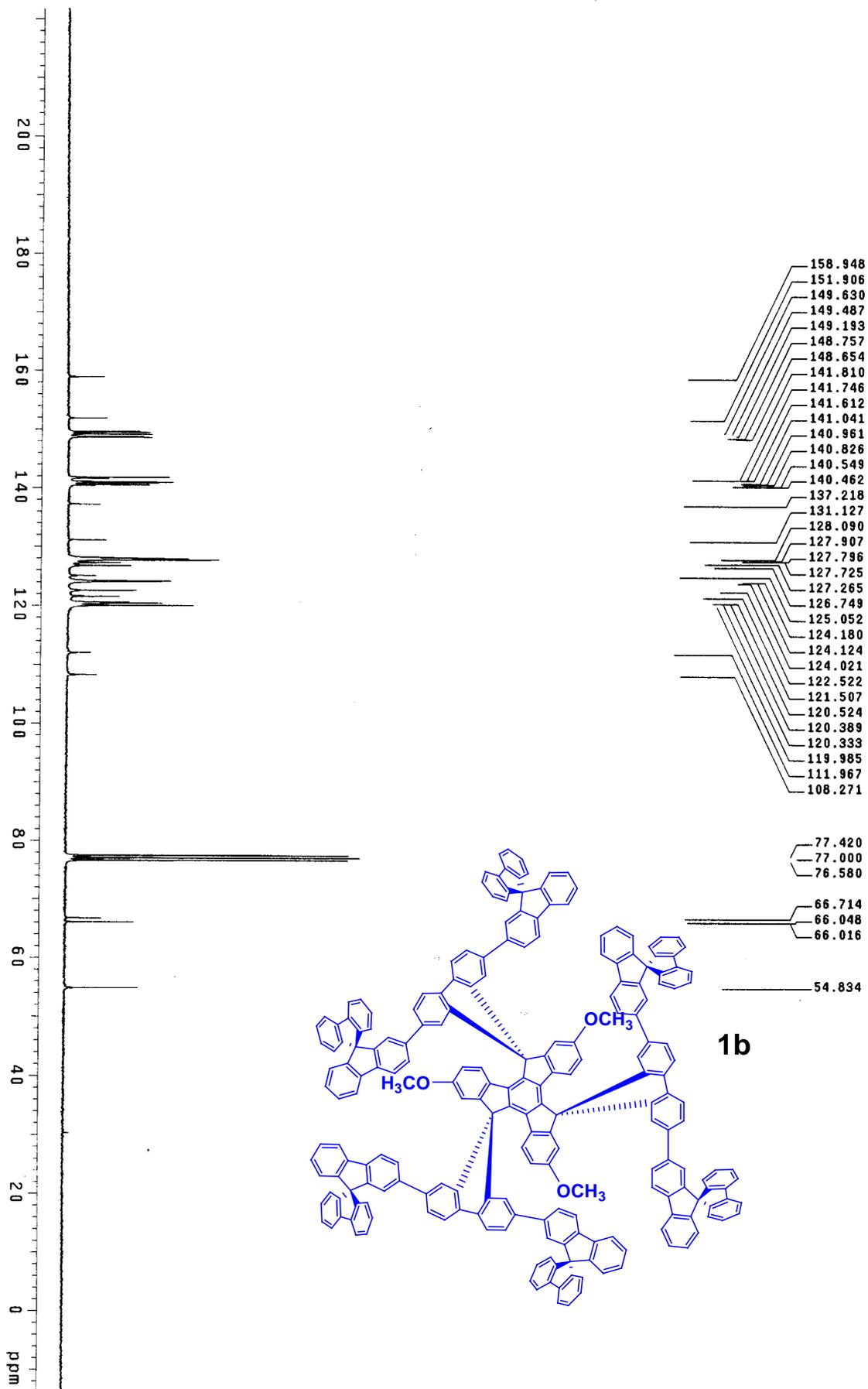


Di as matrix

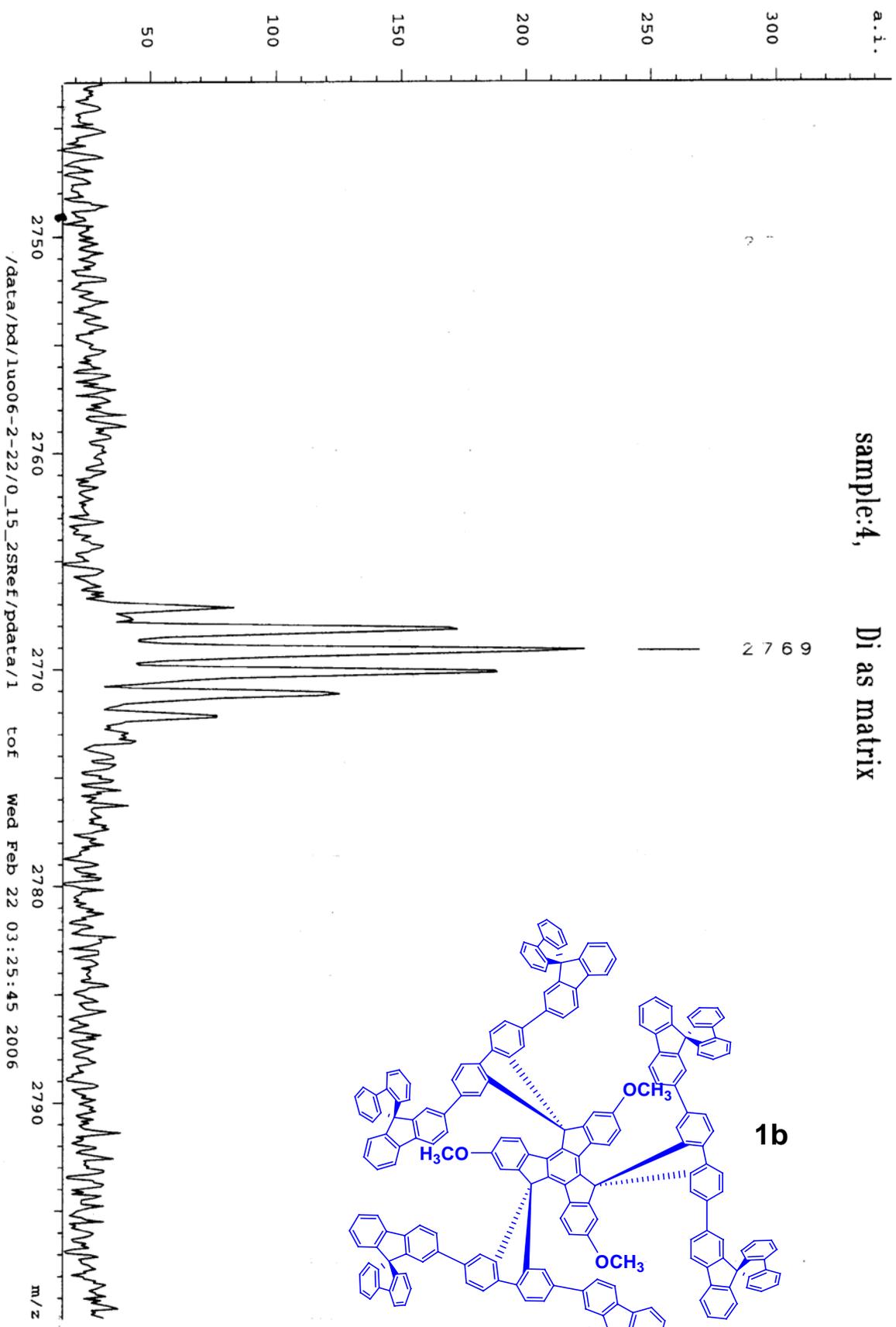


300MHz-SP3

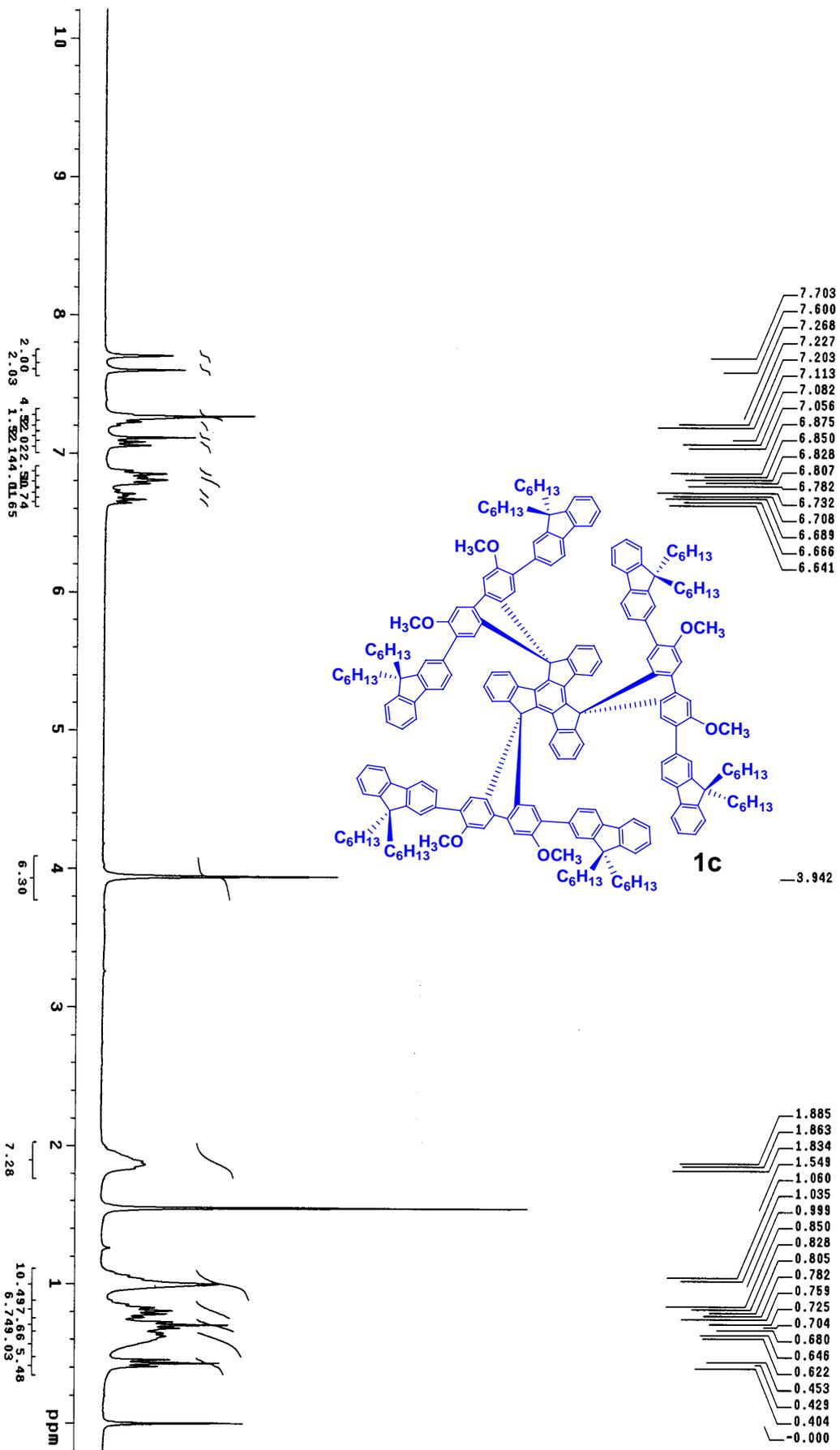


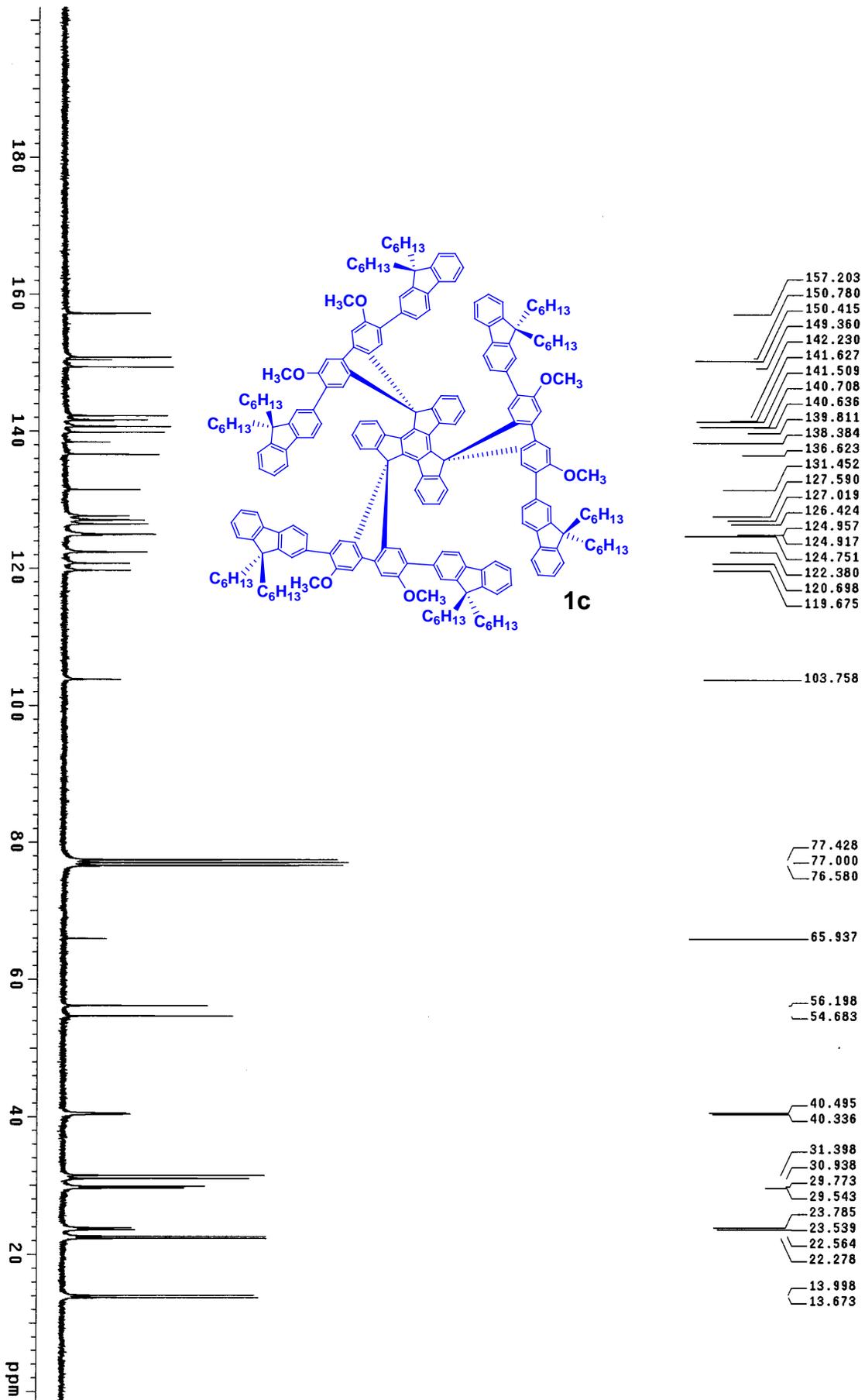


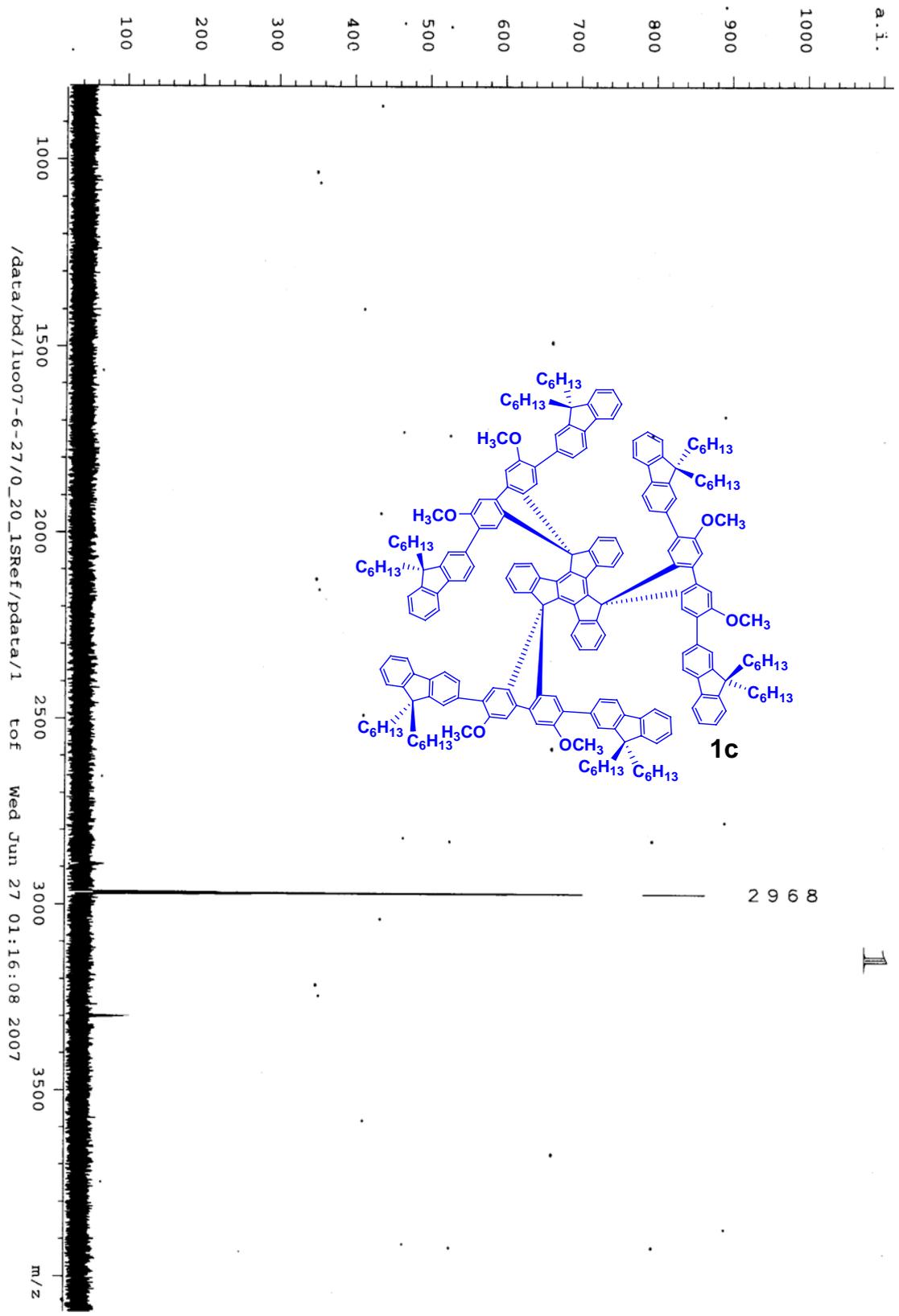
sample:4, Di as matrix



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